

Fig. 2. A stereoview packing diagram of the unit cell. Au atoms are shown as filled circles; P, S and Cl atoms as open circles. H atoms have not been included.

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1,3,7,9-Tetra-*tert*-butyl-2,2,8,8-tetramethyl-5,10-diphenyl-1,3,5,7,9,10-hexaaza-2,8-disila-4,6-distannadispiro[3.1.3.1]decane

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Abstract. $C_{32}H_{58}N_6Si_2Sn_2$, $M_r = 820.41$, monoclinic, $P2_1/c$, $a = 9.361$ (9), $b = 14.006$ (8), $c = 15.814$ (10) Å, $\beta = 105.41$ (6)°, $V = 1999$ (3) Å³, $Z = 2$, $D_x = 1.363$ Mg m⁻³, $\lambda(\text{Ag } K\alpha) = 0.5608$ Å, $\mu = 0.7$ mm⁻¹, $F(000) = 840$, $T = 292$ (1) K, final $R = 0.038$ for 2042 unique observed diffractometer data and 191 variables. The central part of the centrosymmetric molecule is a four-membered Sn–N–Sn–N ring [Sn–N: 2.032 (5), 2.041 (5) Å, N–Sn–N: 80.8 (2)°, Sn–N–Sn: 99.2 (2)°]. A phenyl group is bound to each N atom of this ring and each Sn atom of this central ring is also a member of an outer four-membered Sn–N–Si–N ring [Sn–N: 2.005 (5), 2.027 (5), Si–N: 1.746 (5), 1.733 (5) Å, N–Sn–N: 76.8 (2)°, Sn–N–Si: 95.6 (2), 95.3 (2)°, N–Si–N: 92.2 (2)°]. The dihedral angle between the plane through the central ring and the least-squares plane through the outer ring is 90.3 (3)°. There are no short intermolecular contacts. The compound is the first and unexpected example of this linear dispiro ring system.

Experimental. The substance (Neumann & Obloh, 1986) has been obtained from the known stannylene (Veith, 1975) and phenyl azide at 298 K. Colourless crystals from boiling THF by slow cooling to 273 K.

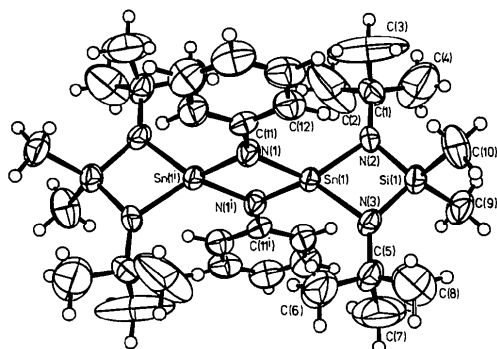
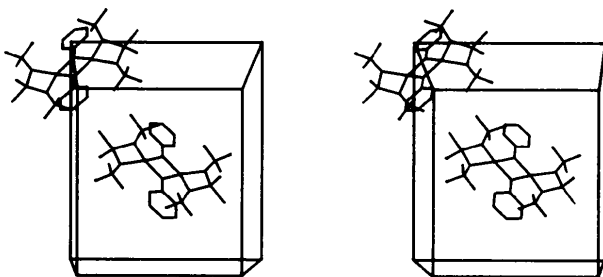
Crystal size 0.45 × 0.18 × 0.16 mm, enclosed in a capillary, $\omega/2\theta$ scan, scan speed 1.8–6.7° min⁻¹ in θ , Nonius CAD-4 diffractometer, graphite-

monochromated Ag $K\alpha$; lattice parameters from least-squares fit with 25 reflections up to $2\theta = 24.3^\circ$; four standard reflections recorded every 2.5 h, only random deviations; 8044 reflections measured; $1 \leq \theta \leq 20^\circ$, $-11 \leq h \leq 11$, $0 \leq k \leq 17$, $-19 \leq l \leq 19$, max. $(\sin\theta)/\lambda = 0.61$ Å⁻¹; after averaging ($R_{\text{int}} = 0.033$): 3961 unique reflections, 2042 with $I > 1.96\sigma(I)$; Lorentz-polarization correction and absorption correction *via* ψ scans; systematic absences $(h0l)$ $l = 2n + 1$, $(0k0)$ $k = 2n + 1$; space group $P2_1/c$; structure solution *via* direct methods, ΔF syntheses and full-matrix least-squares refinement with anisotropic temperature factors for all non-H atoms and a common isotropic temperature factor for H atoms, which were placed in geometrically calculated positions (C–H 0.95 Å); refinement on F with 2042 reflections and 191 refined parameters; $w = 4F_o^2/[\sigma^2(F_o^2) + 0.08F_o^2]$; $S = 0.80$, $R = 0.038$, $wR = 0.046$, $(\Delta/\sigma)_{\text{max}} = 0.02$; no extinction correction; largest peak in final ΔF map ± 0.5 (2) e Å⁻³; complex neutral-atom scattering factors from *International Tables for X-ray Crystallography* (1974); programs: Enraf–Nonius *SDP* (Frenz, 1981), *ORTEPII* (Johnson, 1976), *MULTAN80* (Main *et al.*, 1980), *POPI* (van de Waal, 1976).

The molecule and the numbering scheme are shown in Fig. 1 and a stereoscopic view of the unit cell in Fig. 2. Positional parameters and the equivalent values of the anisotropic temperature factors for the non-H

Table 1. Atomic coordinates and equivalent isotropic thermal parameters ($\text{\AA}^2 \times 10^3$)
$$U_{eq} = \frac{1}{6\pi^2} \sum_i \sum_j \beta_{ij} \mathbf{a}_i \cdot \mathbf{a}_j$$

	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{eq}
Sn(1)	0.45665 (5)	-0.10017 (3)	0.02691 (3)	40
Si(1)	0.3708 (2)	-0.2804 (1)	0.0696 (1)	51
N(1)	0.6083 (5)	0.0050 (4)	0.0729 (4)	45
N(2)	0.3252 (6)	-0.1638 (4)	0.0916 (4)	50
N(3)	0.4967 (6)	-0.2410 (3)	0.0144 (4)	47
C(1)	0.2178 (8)	-0.1217 (5)	0.1330 (5)	64
C(2)	0.269 (1)	-0.0298 (9)	0.171 (1)	242
C(3)	0.077 (1)	-0.108 (1)	0.068 (1)	225
C(4)	0.185 (1)	-0.185 (1)	0.1977 (9)	195
C(5)	0.6113 (9)	-0.2863 (5)	-0.0182 (5)	60
C(6)	0.657 (1)	-0.2237 (7)	-0.0803 (7)	135
C(7)	0.747 (1)	-0.306 (1)	0.057 (1)	165
C(8)	0.559 (1)	-0.3792 (8)	-0.0581 (9)	160
C(9)	0.455 (1)	-0.3545 (6)	0.1683 (6)	76
C(10)	0.212 (1)	-0.3491 (6)	0.0006 (7)	90
C(11)	0.7229 (6)	0.0078 (4)	0.1500 (4)	43
C(12)	0.8229 (8)	0.0847 (5)	0.1677 (5)	58
C(13)	0.9365 (9)	0.0859 (6)	0.2446 (5)	70
C(14)	0.9547 (9)	0.0141 (7)	0.3047 (5)	77
C(15)	0.859 (1)	-0.0620 (7)	0.2884 (5)	77
C(16)	0.7420 (8)	-0.0647 (6)	0.2123 (5)	58

Fig. 1. General view of the molecule [(i) corresponds to the symmetry operation: $1-x, -y, -z$].Fig. 2. Stereoscopic view of the unit cell (*b* axis horizontal, *c* axis vertical).

atoms are given in Table 1.* Bond lengths, bond angles, least-squares planes and dihedral angles are given in Table 2.

Related literature. Veith (1981).

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* Lists of H-atom coordinates, anisotropic thermal parameters and structure-factor amplitudes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43536 (26 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. Bond distances (\AA), angles ($^\circ$), least-squares planes and dihedral angles ($^\circ$)

Sn(1)—N(1 ¹)	2.032 (5)	C(1)—C(2)	1.45 (1)
Sn(1)—N(1)	2.041 (5)	C(1)—C(3)	1.45 (1)
Sn(1)—N(2)	2.005 (5)	C(1)—C(4)	1.45 (2)
Sn(1)—N(3)	2.027 (5)	C(5)—C(6)	1.46 (1)
Si(1)—N(2)	1.746 (5)	C(5)—C(7)	1.52 (1)
Si(1)—N(3)	1.733 (5)	C(5)—C(8)	1.47 (1)
Si(1)—C(9)	1.864 (9)	C(11)—C(12)	1.405 (8)
Si(1)—C(10)	1.863 (9)	C(12)—C(13)	1.39 (1)
N(1)—C(11)	1.393 (7)	C(13)—C(14)	1.36 (1)
N(2)—C(1)	1.462 (8)	C(14)—C(15)	1.37 (1)
N(3)—C(5)	1.454 (8)	C(15)—C(16)	1.39 (1)
		C(16)—C(11)	1.39 (1)
N(1)—Sn(1)—N(1 ¹)	80.8 (2)	N(2)—C(1)—C(2)	110.6 (6)
N(1)—Sn(1)—N(2)	127.0 (2)	N(2)—C(1)—C(3)	110.0 (8)
N(1)—Sn(1)—N(3)	127.4 (2)	N(2)—C(1)—C(4)	111.6 (7)
N(1 ¹)—Sn(1)—N(2)	126.2 (2)	C(2)—C(1)—C(3)	108 (1)
N(1 ¹)—Sn(1)—N(3)	126.0 (2)	C(2)—C(1)—C(4)	111.3 (9)
N(2)—Sn(1)—N(3)	76.8 (2)	C(3)—C(1)—C(4)	105.1 (8)
Sn(1)—N(1)—Sn(1 ¹)	99.2 (2)	N(3)—C(5)—C(6)	110.4 (6)
Sn(1)—N(2)—C(1)	129.3 (4)	N(3)—C(5)—C(7)	110.1 (7)
C(11)—N(1)—Sn(1 ¹)	131.5 (4)	N(3)—C(5)—C(8)	110.3 (6)
Sn(1)—N(2)—Si(1)	95.6 (2)	C(6)—C(5)—C(7)	107.7 (7)
Sn(1)—N(2)—C(1)	129.6 (4)	C(6)—C(5)—C(8)	111.4 (8)
Si(1)—N(2)—C(1)	134.4 (4)	C(7)—C(5)—C(8)	106.9 (9)
Sn(1)—N(3)—Si(1)	95.3 (2)	N(1)—C(11)—C(12)	120.9 (6)
Sn(1)—N(3)—C(5)	129.3 (4)	N(1)—C(11)—C(16)	121.5 (5)
Si(1)—N(3)—C(5)	134.8 (4)	C(12)—C(11)—C(16)	117.6 (6)
N(2)—Si(1)—N(3)	92.2 (2)	C(11)—C(12)—C(13)	120.0 (7)
N(2)—Si(1)—C(9)	115.1 (3)	C(12)—C(13)—C(14)	121.8 (7)
N(2)—Si(1)—C(10)	113.6 (3)	C(13)—C(14)—C(15)	119.2 (7)
N(3)—Si(1)—C(9)	114.1 (3)	C(14)—C(15)—C(16)	120.4 (8)
N(3)—Si(1)—C(10)	114.5 (4)	C(15)—C(16)—C(11)	121.0 (7)
C(9)—Si(1)—C(10)	107.2 (4)		

Equation of the plane

(*x* along **a**; *y* in plane *ab*;

z along **c***)

No.	Plane through atoms	ψ^2
1	Sn(1), Sn(1 ¹), N(1), N(1 ¹)	0
2	Sn(1), N(2), N(3), Si(1)	1130
3	C(11), C(12), C(13), C(14), C(15), C(16)	3

Dihedral angles: 1,2 90.3; 1,3 3.9; 2,3 89.4 ($\sigma \sim 0.3$)

(i) corresponds to the symmetry operation: $1-x, -y, -z$.

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trans-Dichloro(tricyclohexylphosphino)(triethylphosphino)platinum(II)

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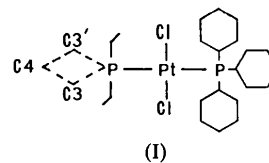
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Abstract. [PtCl₂P(C₂H₅)₃P(C₆H₁₁)₃], $M_r = 664.59$, orthorhombic, $Pcab$, $a = 12.284$ (5), $b = 19.409$ (6), $c = 23.918$ (6) Å, $V = 5702.5$ Å³, $Z = 8$, $D_x = 1.548$ Mg m⁻³, $\lambda(\text{Mo } K\alpha) = 0.71073$ Å, $\mu = 5.28$ mm⁻¹, $F(000) = 2672$, $T = 291$ K, final $R = 0.029$ for 1988 unique observed reflections with $I > 3\sigma(I)$. The Pt atom has square-planar coordination, with the P–Pt–C angle involving the P(Et)₃ ligand distorted due to the bulk of the tricyclohexylphosphine ligand. The phosphine ligands are oriented *trans* across the square plane of coordination. One of the methylene carbon atoms of the triethylphosphine is disordered, with each site having 50% population. Selected metrical details are $\langle \text{Pt–Cl} \rangle = 2.304$ (1), $\langle \text{Pt–P} \rangle = 2.333$ (42), and $\langle \text{C–C} \rangle = 1.523$ (12) Å.

Experimental. Crystals of title compound (I) obtained from Dr H. C. Clark of the University of Guelph; colorless rectangular-shaped crystal, $0.19 \times 0.28 \times 0.47$ mm, glass fiber mount, Enraf-Nonius CAD-4F four-circle diffractometer, graphite-monochromatized Mo $K\alpha$ radiation; unit-cell dimensions and systematic absences $0kl$, $l = 2n + 1$, $h0l$, $h = 2n + 1$, $hk0$, $k = 2n + 1$; cell constants from setting angles of 25 reflections ($4.0 \leq \theta \leq 9.8^\circ$); correction for Lorentz, polarization effects, empirical absorption correction (North, Phillips & Mathews, 1968), $t(\text{min})-t(\text{max}) = 0.506-0.997$; 291 K; intensity data collected by $\omega-2\theta$ scan technique, variable scan rate of $0.38-3.35^\circ\text{min}^{-1}$, intensities of two check reflections (464 and 373) measured every 2 h revealed only random deviations (< 1%) from mean intensities; 3539 unique

reflections collected ($3 < 2\theta < 45^\circ$, $h: 0 \rightarrow 13$, $k: 0 \rightarrow 20$, $l: 0 \rightarrow 25$), 1988 reflections with $I > 3\sigma(I)$ used in solution and refinement of the structure using *SDP/VAX* (Enraf-Nonius, 1983); Pt atom located by *MULTAN11/82* (Main, 1982), remainder of non-hydrogen atoms by difference Fourier technique; H atoms calculated and included at fixed positions with $B = 5.0$ Å²; full-matrix least-squares refinement on F yielded $R = 0.0289$, $wR = 0.0278$, $w = \sigma^{-2}(F_o)$ with $\sigma(F_o) = \{I_p + I_b + [0.03(I_p - I_b)]^2\}^{1/2}$, where I_p and I_b are the peak and background counts respectively, $S = 0.928$, maximum shift-to-e.s.d. ratio of a parameter in final least-squares cycle was 0.006; maximum positive and minimum negative densities on final difference Fourier map 1.4 (1) and 0.6 (1) e Å⁻³ in vicinity of Pt atom; scattering factors and anomalous-dispersion terms taken from *International Tables for X-ray Crystallography* (1974).



The final atomic parameters are given in Table 1,† with interatomic distances and angles presented in

† Lists of anisotropic thermal parameters, H-atom coordinates, dihedral angles, bond lengths and angles, structure factors, and illustrations of the ligand profile about the phosphine P atoms have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43499 (31 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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